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FEASIBILITY OF MINIATURIZING A HEATER FOR A THIN-FILM OXYGEN PARTIAL-PRESSURE SENSOR

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16. Abstract

The feasibility of using a miniature heater for a zinc oxide thin-film oxygen partial-pressure sensor was investigated. A silicon heater 0.25 millimeter square by 13 millimeter long was used to obtain the desired operating temperature for the zinc oxide film, 650 K. Experimental data are presented for the resistance-temperature characteristics, the temperature distribution, and power requirements of the heater. Results show the temperature of the heater can be determined by the change in resistance of the heater at its operating temperature. This work has also shown that this heater concept is feasible, but more effort will be required to deposit the zinc oxide film on the heater surface.

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SUMMARY

The zinc oxide thin-film technique for sensing oxygen partial pressure is a promising sensing method. The feasibility of using a miniaturized heater in order to reduce the size and power requirements of the sensor was investigated.

Heater power is minimized by reducing the heater diameter as much as practical. A bar of silicon 13 millimeters long and 0.25 millimeter square was selected for the heating element. The surface temperature of the heater was measured by placing a miniature thermocouple on the surface of the bar with a three-axis micropositioner. The resistance and power requirements of the heater were measured as a function of its maximum surface temperature, and the surface temperature distribution was determined.

The results indicated that the use of a small-diameter heater for a zinc oxide oxygen sensor is feasible and will reduce the oxygen sensor size and power requirements. It was also shown that the temperature of the heater in an actual sensor can be determined by measuring its average resistance.

INTRODUCTION

The selection of a satisfactory cabin atmosphere for manned space vehicles poses a number of conflicting problems. Current indications point toward the use of an oxygen-inert gas atmosphere for long-duration space flights. This atmosphere requires the use of an oxygen partial-pressure sensor.

The requirements for space flight such as minimum weight, size, and power consumption, together with the necessity for long-term accuracy and stability, eliminate most sensing methods from consideration. Present sensors utilizing paramagnetic,

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polarographic, ultraviolet absorption, and solid electrolyte techniques have fundamental limitations to further miniaturization. The zinc oxide thin-film sensor, however, has shown the greatest promise of being successfully miniaturized and also of meeting flight requirements. Under Contract NAS1-7087, Research Triangle Institute conducted an investigation of zinc oxide thin films for sensing oxygen and constructed a laboratory model using this technique (ref. 1). Their working model consisted of a nichrome wire heating element surrounded by a hollow cylindrical substrate on which a zinc film was deposited and then oxidized at 873 K. This whole unit was then enclosed in an insulating housing similar to a miniature thermos bottle. This arrangement resulted in a sensor weighing about 30 grams, occupying approximately 30 cubic centimeters of volume, and requiring 5 to 6 watts of power for operation at 650 K. Inherent problems with a wire-wound heater place a lower limit on the physical size and power requirements of the sensor.

The purpose of the research reported herein is to indicate the feasibility of overcoming the size and power limitation of a wire-wound heater by fabricating a heater from a single length of material. This concept would allow the construction of an integrated heater, heater temperature sensor, and oxygen sensor in one miniaturized unit. The heater power would be minimized by minimizing the heater surface area.

The objectives of this research are as follows:

(1) To select a heater material with proper resistivity

(2) To develop reliable electrical connections to the heater

(3) To develop a method of measuring the heater temperature

(4) To minimize heater power by making dimensions as small as practical

Two silicon heaters were fabricated and electrical contacts were made to the heaters. Tests were conducted to determine the resistance as a function of temperature characteristics, temperature distribution, and heater power requirements of the silicon heaters.

APPARATUS AND METHODS

Zinc Oxide Thin-Film Technique

The zinc oxide sensing technique makes use of the sensitivity to oxygen partial pressure of zinc oxide films in the thickness range of about 500 to 1000 angstroms. The oxygen acts as a doping agent to the zinc oxide semiconductor.

A diagram of the first laboratory model zinc oxide thin-film oxygen sensor from reference 1 is shown in figure 1. This model consisted of a nichrome wire-wound heater, a platinum resistance temperature sensor, and a tubular substrate on which the zinc oxide film was deposited.

The temperature dependence of zinc oxide electrical conductivity as determined in reference 1 is shown in figure 2. Operation of the sensor at the 650 K minimum or the 475 K maximum would result in minimum temperature sensitivity, minimum required temperature control accuracy, and minimum effect of a temperature gradient along the required heater. The largest sensitivity to oxygen is at 650 K; in addition, the response time at the minimum is considerably faster as compared with that at the maximum. Thus, the best operating point can be seen to be at 650 K. A more detailed treatment of this technique is given in reference 1.

Selection of Heater Material

The following parameters were chosen as the initial design criteria for a heater consisting of a single short length of material: (1) voltage less than 100 volts, (2) current less than 100 milliamperes, and (3) surface area and power requirement significantly less than those of currently available heaters.

For the purposes of this feasibility study, a piece of n-type silicon 13 millimeters long by 0.25 millimeter square was selected. In order to make use of the constant temperature coefficient of log resistance for the intrinsic region to sense the operating temperature, a room-temperature resistivity of approximately 1 ohm-centimeter was chosen. As a first approximation, the whole bar is assumed to be at 650 K and the heater resistance should then be approximately 2500 ohms.

Temperature Characteristics of Silicon

Figure 3 (adapted from ref. 2) shows a family of curves for the temperature dependence of the resistivity of single crystal n-type silicon for different doping levels. The intrinsic curve is independent of the room-temperature doping level. However, the temperature at which conduction transfers from extrinsic to intrinsic is a function of the doping level.

Test Apparatus

The dimensions of the silicon bar heater and ceramic mounting support used for this study are shown in figure 4. The electrical contacts were painted on the bar, down the mounting frame, and across the ceramic base where contact was made to copper wires.

As shown in figure 5, the heater mounting frame was placed on the table of a three-axis micropositioner. A microminiature thermocouple was rigidly mounted and with the aid of a 120 power stereo zoom microscope, the thermocouple was placed against the surface of the heater.

Figure 6 shows the electrical details of the test apparatus. The clip-on milliammeter has an accuracy of ±3 percent, the voltmeter has an accuracy of ±1 percent, and the cold junction reference has an accuracy of $\pm\frac{1}{4}$ K.

The temperature-measuring thermocouple consisted of a copper constantan junction formed with 0.025-millimeter-diameter wires. The wires were run through a continuous solid quartz insulator and moisture barrier that was sheathed in a 0.76-millimeter-diameter stainless-steel tube. The wires protrude beyond the tube and are formed into a junction of 0.25-millimeter diameter. The thermocouple would definitely conduct some heat from the heater and thus the measured temperature would be less than the actual temperature. This method of temperature measurement is sufficiently accurate for the purposes of this feasibility study since the power required to achieve the operating temperature with no temperature-measurement error would be less than the power required in the experimental situation.

RESULTS AND DISCUSSION

Heater Electrical Contacts

Liquid bright gold paste leads fired on the bar proved to be a successful method of achieving electrical contacts. The gold paste was painted on the ends of the bar, down the ceramic supports, and on to conducting pads on the base. Liquid bright gold contacts have been known to degrade in a few days when operated at elevated temperatures. (See ref. 1, p. 3.) In this particular application no degradation has been noticed over periods as long as 6 months. The ends of the bar where the contacts are located are not at a temperature anywhere near 650 K, which is most likely the reason for the stability.

With the gold contacts, the heater resistance was found to be a function of the heater current. However, at temperatures at the center of the heater greater than 370 K, the contacts become ohmic so that when the center of the heater is at the operating temperature of 650 K, the resistance characteristics will be due primarily to the silicon bar alone.

Measurement of Heater Resistance As a Function of Temperature

In an actual sensor designed to use a heater as small as 0.25 millimeter square, the measurement of heater temperature would be difficult by conventional temperature sensors. If the resistance is calibrated as a function of temperature characteristics of each heater, standard resistance thermometry techniques can be utilized to make the heater serve as its own temperature sensor. Since the heater has a heat sink at each end and an internal heat source from the current flowing through the heater, the heater will have a significant temperature distribution along its length with the maximum temperature occurring theoretically at the midpoint of its length. The method of approach will therefore be to calibrate the total heater resistance against the maximum or midpoint temperature. The zinc oxide thin film will then be deposited along a small portion of the length of the heater and centered about the point of maximum temperature.

Figure 7 indicates the variation of resistivity of a silicon sample with a uniform temperature distribution as a function of temperature. These data were obtained from figure 3, which was taken from reference 2.

Figure 8 shows the experimentally measured dependence of resistance of two different silicon heaters on the maximum heater temperature. The current flowing through the heater was varied and the resistance was calculated from the measured heater current and voltage.

The initial sharp decrease in resistance in figure 8 at room temperature is due to the rectifying contacts on the heater. Once the rectifying contact breaks down, the extrinsic curve for both heaters takes on the shape comparable to the extrinsic region in figure 7. The transfer from extrinsic conduction occurs at approximately 470 K, which is the theoretical value for silicon (ref. 3, p. 209).

At maximum heater temperatures above 470 K, only a portion of the heater goes into intrinsic conduction because of the nonuniform temperature distribution along the heater. For this reason the slope of the experimental data in figure 8 at temperatures above 470 K is approximately one-half that of the similar region of figure 7.

The resistance of bar 1 in figure 8 is significantly higher than that of bar 2. This effect is most likely due to physical differences between the two bars. However, the most repeatable characteristics between the two heaters are the maximum resistance at 470 K and the slope of the curve of resistance as a function of temperature for temperatures above 470 K. These two characteristics can be utilized to determine an approximate operating point for the heater without requiring a calibration curve for the specific heater.

Heater-Surface Temperature Distribution

The measured surface temperature distribution is shown in figure 9. As would be expected for a heater with mounts on each end, the maximum temperature occurs at approximately the middle of the heater.

The variation in surface temperature along the heater will be influenced by the heat loss through the mounts and the electrical connections. The temperature variation could be reduced by reducing these losses, but the electrical connection problems would be increased.

Heater Power Requirements

Because of its small size, the heater can be operated in a completely uninsulated condition. Since silicon has a negative temperature coefficient of resistance in the intrinsic state, the heater must be operated from a constant-current power supply. If the heater is operated from a constant-voltage supply, an unstable condition will result at temperatures about 473 K and the temperature will increase quickly to the melting point for silicon.

Figure 10 shows the current and corresponding voltage required as a function of the maximum heater temperature for a typical heater. A 50-milliampere constant-current power supply capable of 100 volts would be required to power the heater with free convection and no insulation.

Figure 11 shows the power required to heat the heater in the uninsulated condition as a function of maximum temperature. The heater required approximately 2.5 watts of power to reach a maximum temperature of 650 K.

In a final configuration this power requirement should be reduced through the use of reflecting radiation shielding.

CONCLUSIONS

This work has shown that the use of a small-diameter silicon heater for a zinc oxide oxygen sensor is feasible and will result in a significant reduction in size and power requirements.

The temperature of the heater can be determined by measuring its average resistance. A significant variation in the resistance characteristics for different elements has been noted, but the slope of the curve of log resistance as a function of temperature for the operating temperature region is repeatable for different heaters. This result allows an approximate operating point to be easily found for each heater.

Langley Research Center,
National Aeronautics and Space Administration,
Hampton, Va., January 15, 1971.

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3. Azároff, Leonid V.; and Brophy, James J.: Electronic Processes in Materials. McGraw-Hill Book Co., Inc., c.1963.

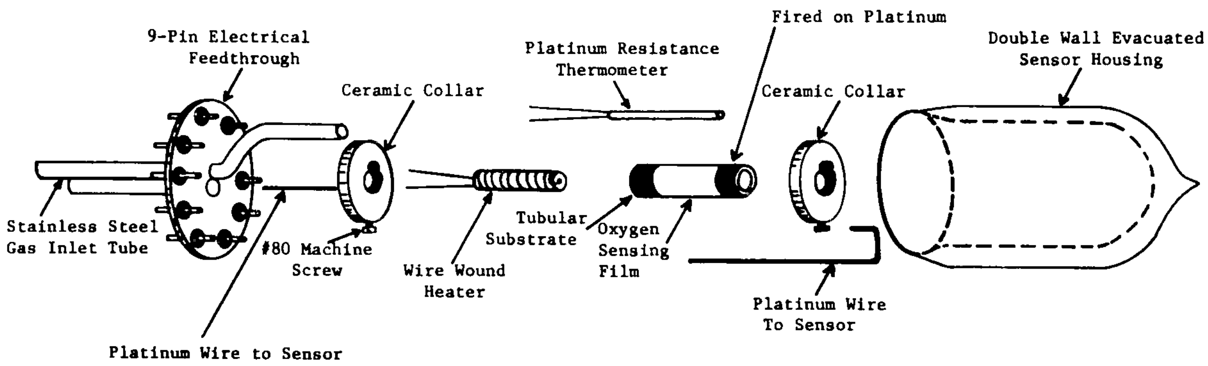

Figure 1.- Details of laboratory model zinc oxide oxygen sensor developed in reference 1 (p. 35).

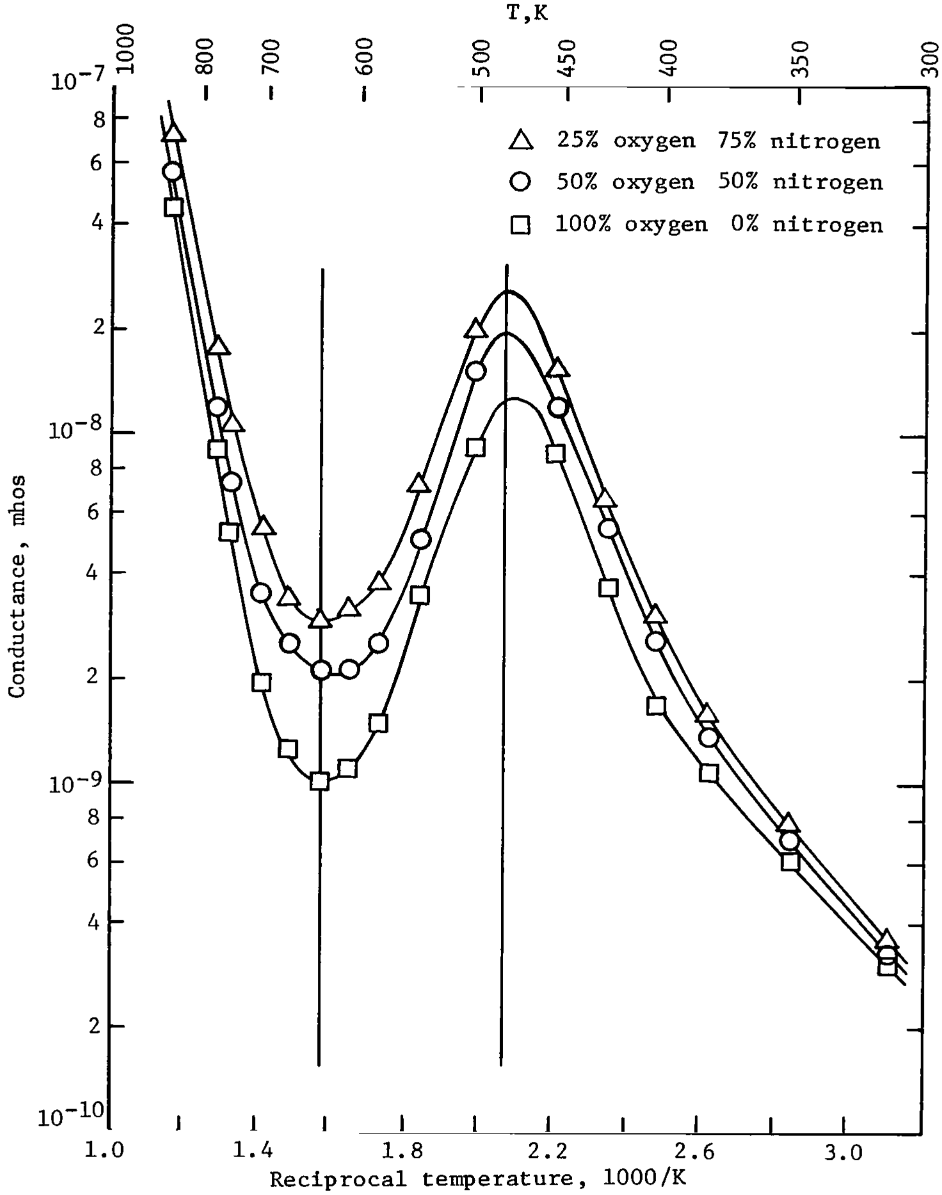

Figure 2.- Electrical conductance of zinc oxide as a function of reciprocal temperature for various partial pressures of oxygen. (From ref. 1, p. 5.)

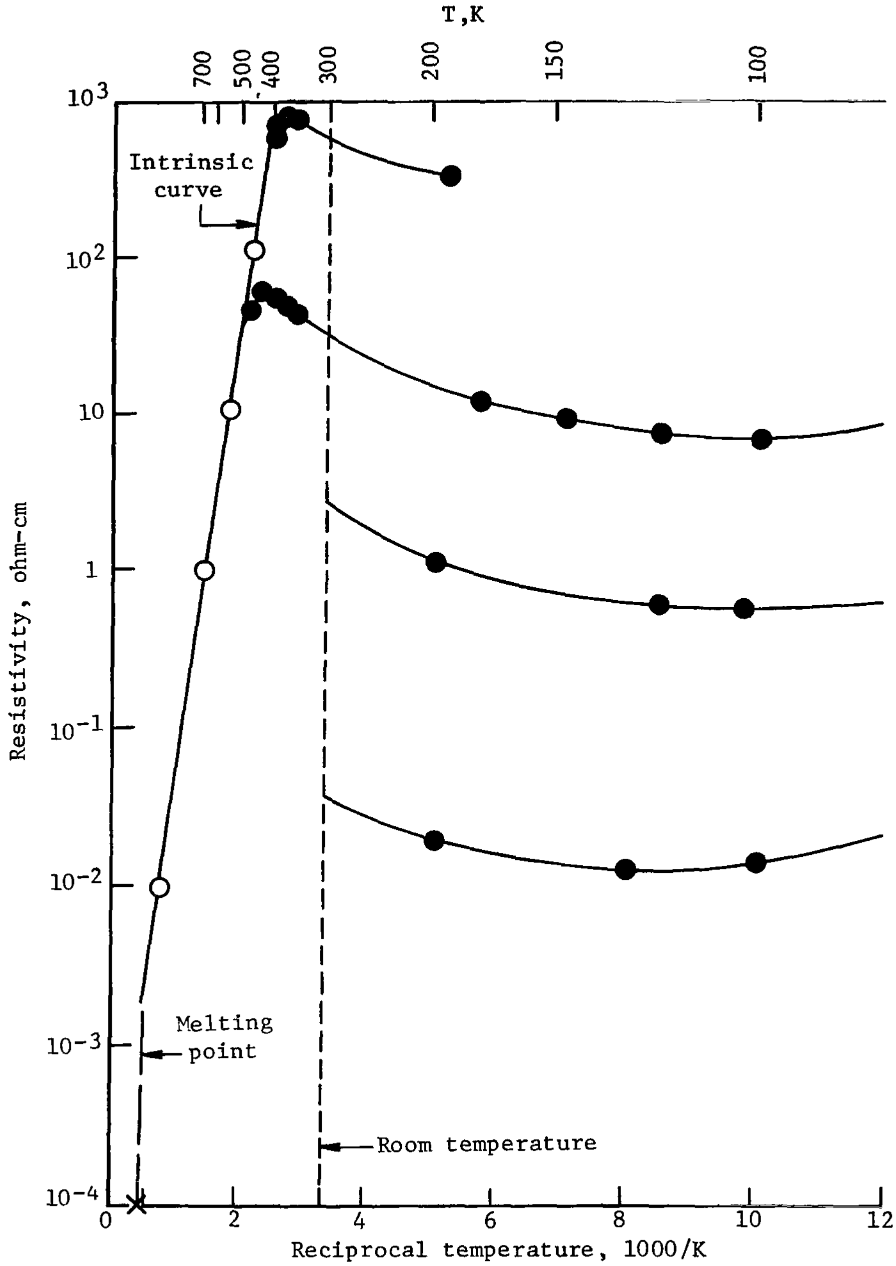

Figure 3.- The temperature dependence of resistivity for n-type single crystal silicon. (Adapted from ref. 2, fig. 2-4.)

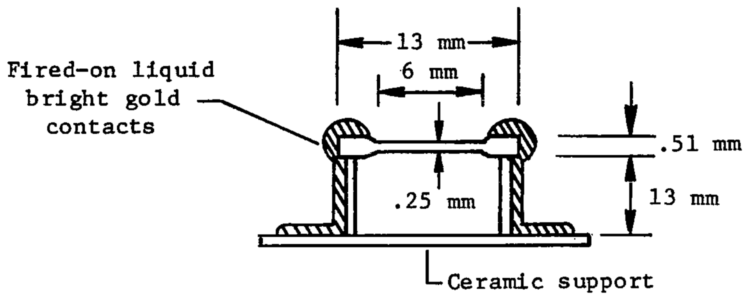

Figure 4.- Details of silicon heater and mounting arrangement.

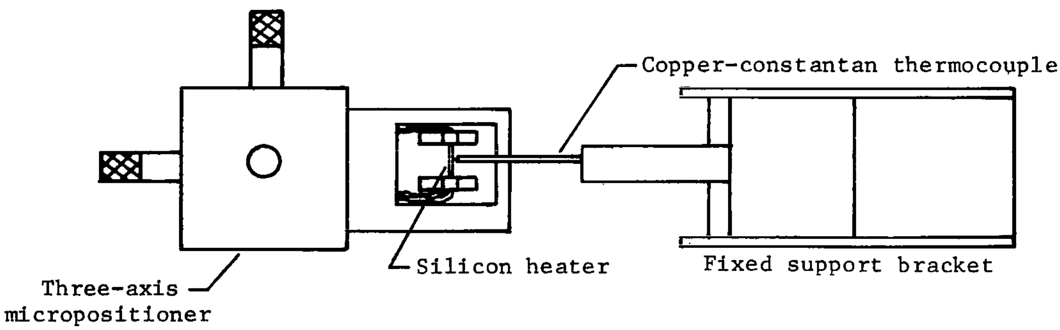

Figure 5.- Mechanical details of test apparatus.

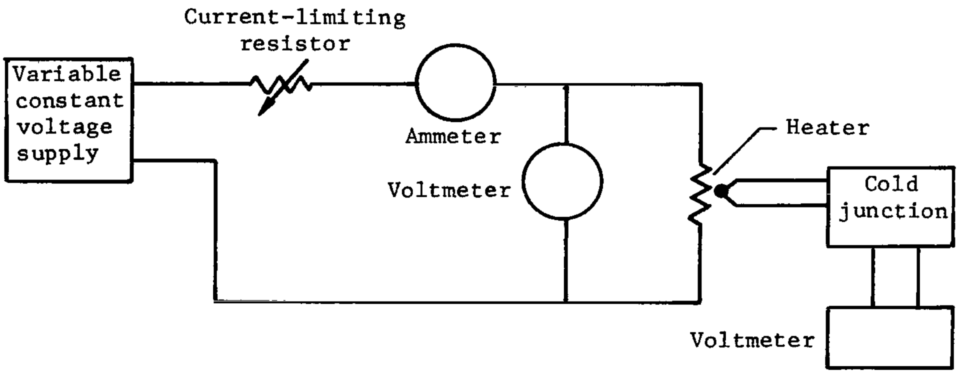

Figure 6.- Electrical details of test apparatus.

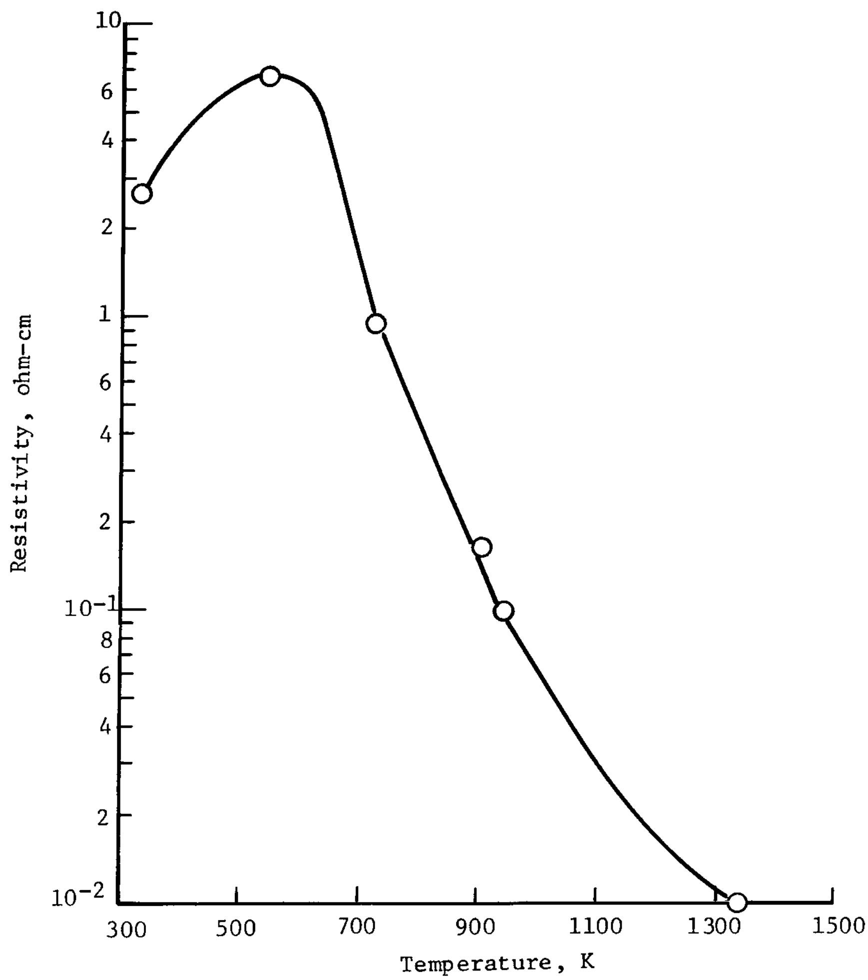

Figure 7.- Resistivity as a function of temperature for silicon.

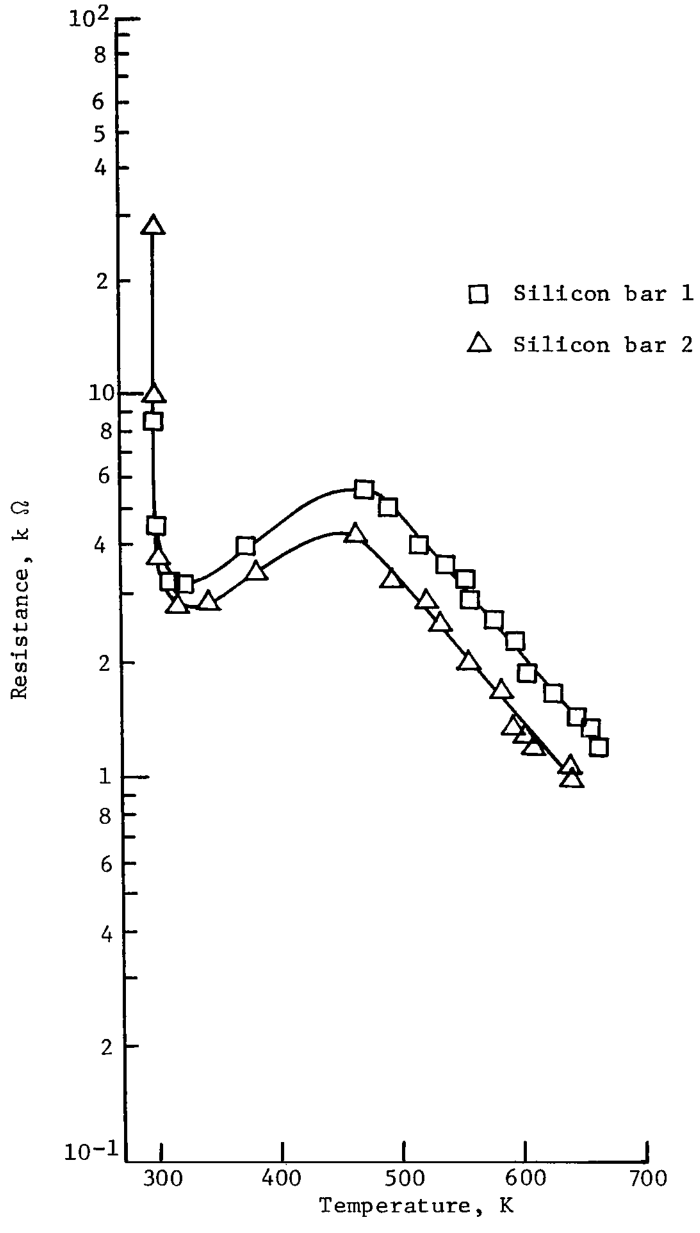

Figure 8.- Experimental resistance as a function of maximum heater temperature characteristics for silicon bar.

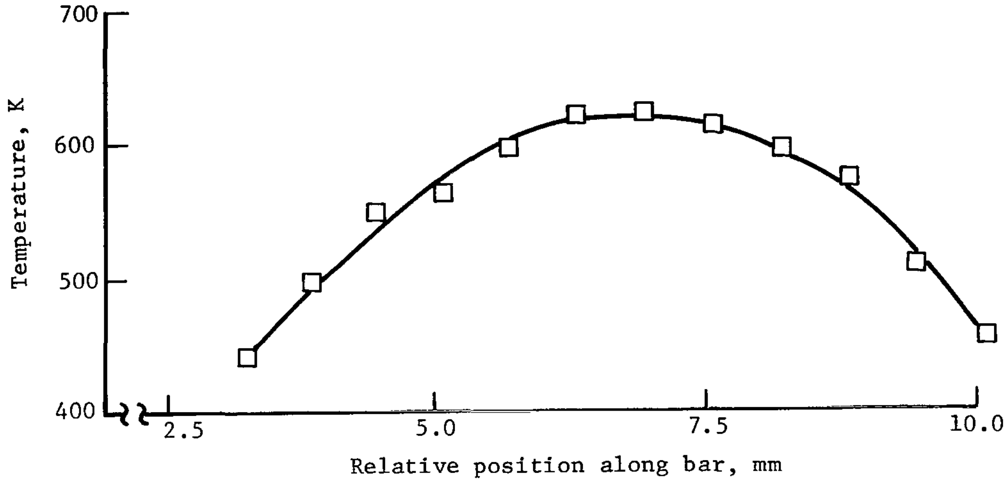

Figure 9.- Surface temperature distribution along heater.

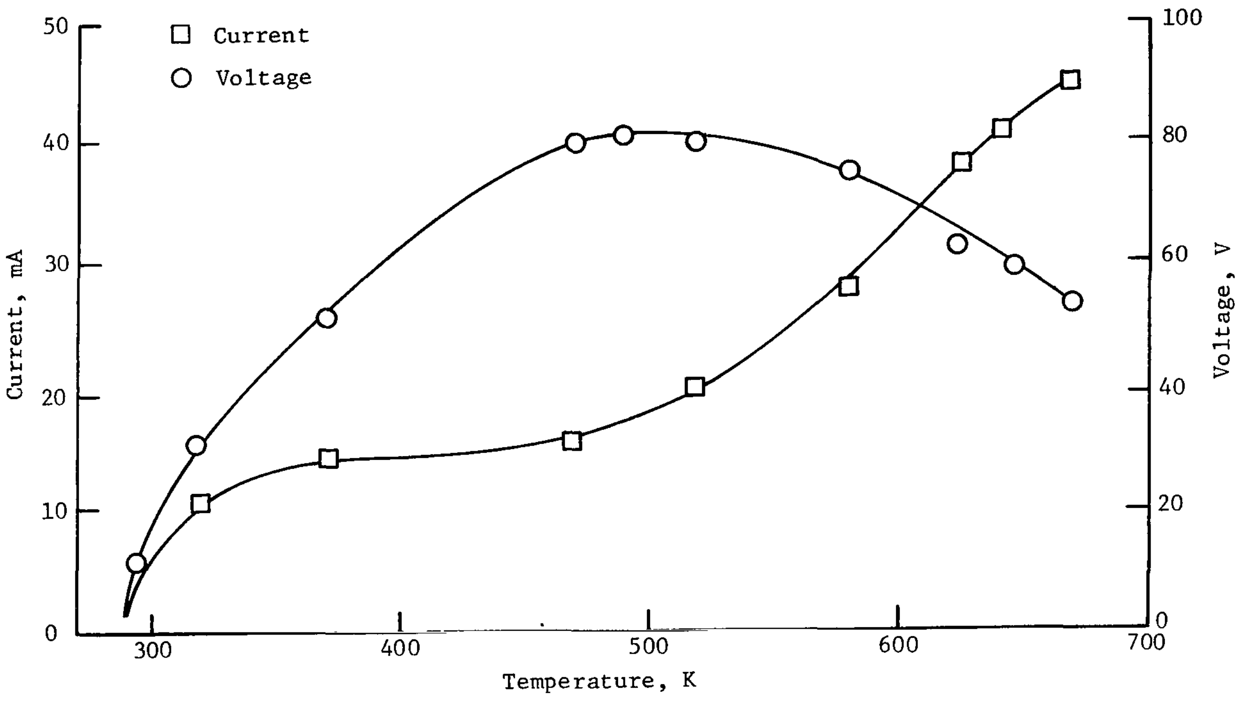

Figure 10.- Current-voltage requirements of the heater.

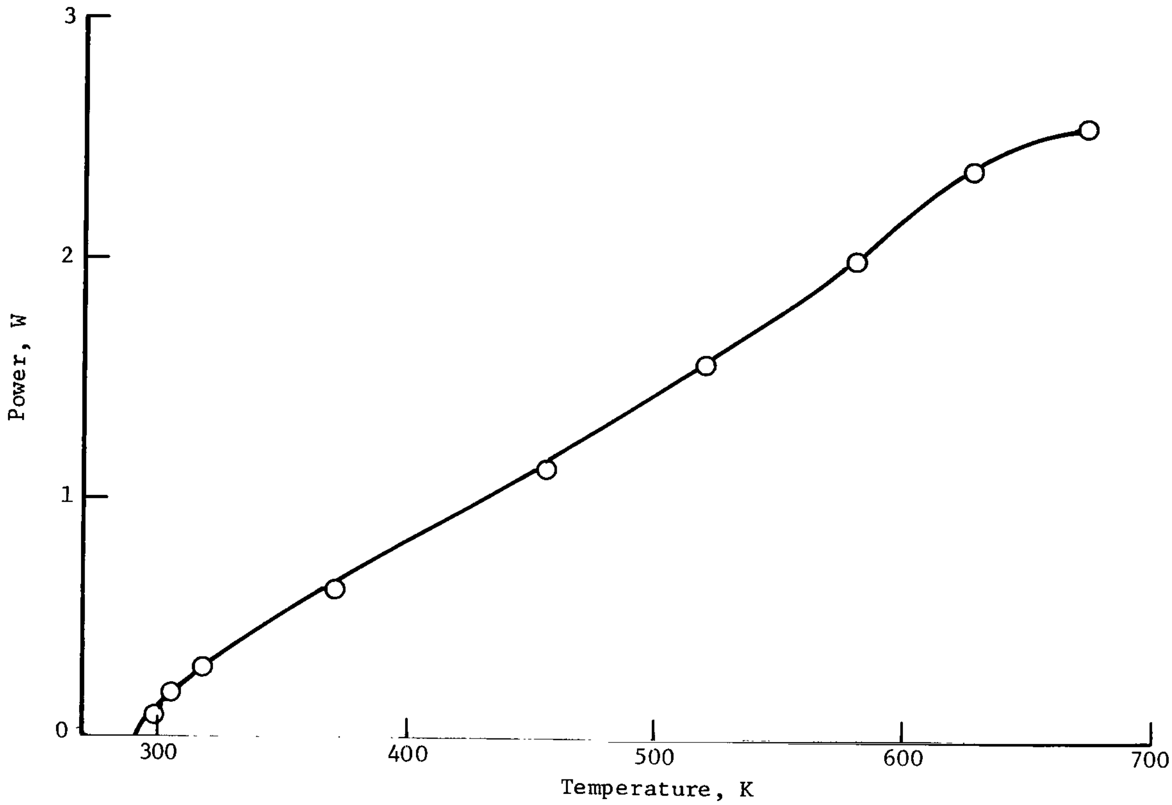

Figure 11.- Power requirements of heater.

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